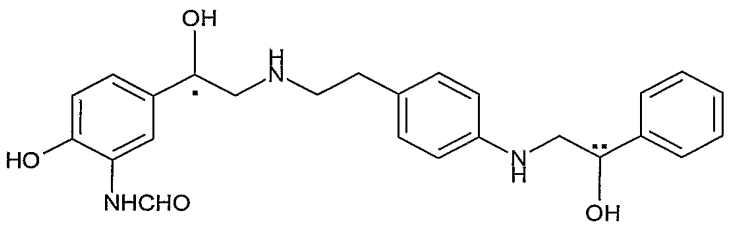
CLAIMS

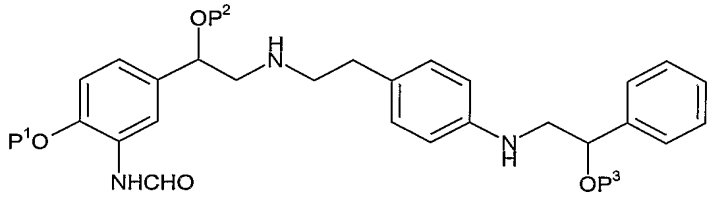
1. A process for preparing a monohydrochloride salt of compound (I)



(I)

wherein *C and **C denote asymmetric carbon atoms,
which process comprises the steps of:

a) contacting a compound of formula (II):



(II)

wherein P^1 represents a hydroxyl protecting group, and P^2 and P^3 each independently represents hydrogen or a protecting group;

with a weak acid, to effect selective protonation;

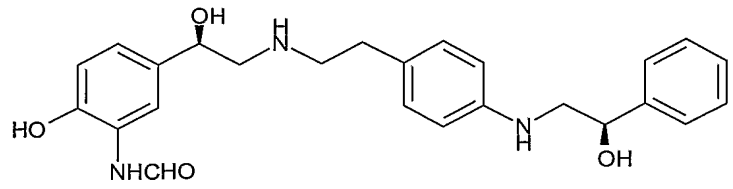
b) contacting the product of (a) with a source of chloride ions, to effect anion exchange;

c) deprotection to remove P^1, and where necessary P^2 and P^3;

d) isolation of compound (I) as the monohydrochloride; and optionally

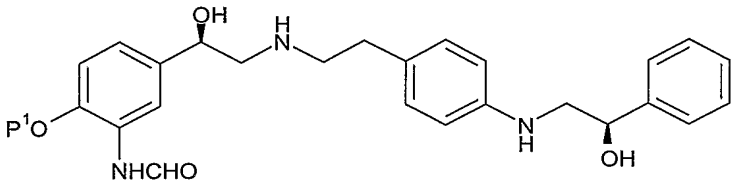
e) crystallisation or recrystallisation of compound (I).

2. A process according to claim 1, wherein the compound of formula (I) is the compound (Ia):



(Ia)

and the compound of formula (II) is the compound (IIa):



(IIa)

wherein P^1 is as defined in claim 1.

3. A process according to claim 1 or claim 2 wherein the weak acid is acetic acid.

4. A process according to any of claims 1 to 3 wherein the group P^1 represents benzyl.

5. A process according to any of claims 1 to 4 wherein the source of chloride ions is sodium chloride.

6. A process according to any of claims 1 to 5 for the preparation of a crystalline monohydrochloride salt of the compound of formula (Ia).

7. A process according to claim 6 wherein the product of said process is characterised by an x-ray powder diffraction pattern in which the peak positions are substantially in accordance with the peak positions of the pattern shown in Fig. 1.

8. Crystalline (Ia) monohydrochloride which is characterised by a differential scanning calorimetry trace which shows an absence of discernable endothermic features below about 125°C.

9. Crystalline (Ia) monohydrochloride according to claim 8 which is characterised by a differential scanning calorimetry trace which shows an absence of discernable endothermic features below about 125°C, and an onset of significant endothermic heat flow at about 229°C.

10. Crystalline (Ia) monohydrochloride according to claim 8 or claim 9 which is characterised by a differential scanning calorimetry trace which shows an absence of discernable endothermic features below about 125°C, two or more minor endothermic events between about 130°C and about 180°C and an onset of significant endothermic heat flow at about 229°C.

11. Crystalline (Ia) monohydrochloride according to claim 10 wherein said minor endothermic events occur at about 133°C, at about 151°C and at about 170°C.

12. Form 2 crystalline (Ia) mononhydrochloride in substantially pure form.

13. A process for obtaining Form 2 crystalline (Ia) monohydrochloride in substantially pure form which process comprises:

Ba) Forming a mixture of *N*-{2-[4-*((R)*-2-hydroxy-2-phenylethylamino)phenyl]ethyl}-(*R*)-2-hydroxy-2-(3-formamido-4-hydroxyphenyl) ethylamine monohydrochloride in an aqueous organic solvent, by contacting said monohydrochloride with said solvent and heating in a range from about 60°C to about 70°C, for example about 65°C;

Bb) Adjusting the temperature of said mixture in the range from about 52°C to about 58°C; for example about 55°C;

Bc) Seeding said mixture with Form 2 crystals;

Bd) cooling said mixture to a temperature in the range from about 15°C to 25°C;

Be) heating said mixture to a temperature in the range from about 47°C to about 52°C, for example about 50°C;

Bf) repeating steps Bd) and Be) to obtain the desired Form 2.

14. A method for the prophylaxis or treatment of a clinical condition in a mammal, such as a human, for which a selective β_2-adrenoreceptor agonist is indicated, which comprises administration of a therapeutically effective amount of Form 2 crystalline (Ia) monohydrochloride.

15. Form 2 crystalline (Ia) monohydrochloride for use in medical therapy.

16. The use of Form 2 crystalline (Ia) monohydrochloride in the manufacture of a medicament for the prophylaxis or treatment of a clinical condition for which a selective β_2-adrenoreceptor agonist is indicated.

17. A pharmaceutical formulation comprising Form 2 crystalline (Ia) monohydrochloride and a pharmaceutically acceptable carrier or excipient, and optionally one or more other therapeutic ingredients.

18. A combination comprising Form 2 crystalline (Ia) monohydrochloride and one or more other therapeutic ingredients.

19. A combination according to claim 17 wherein the other therapeutic ingredient is a PDE4 inhibitor or an anticholinergic or a corticosteroid.

20. A combination according to either of claims 17 or 18 comprising Form 2 crystalline (Ia) monohydrochloride and 6α,9α-difluoro-17α-[(2-furanylcarbonyl)oxy]-11β-hydroxy-16α-methyl-3-oxo-androsta-1,4-diene-17β-carbothioic acid *S*-fluoromethyl ester.

21. A combination according to either of claims 17 or 18 comprising Form 2 crystalline (Ia) monohydrochloride and 6α,9α-difluoro-11β-hydroxy-16α-methyl-17α-[(4-methyl-1,3-thiazole-5-carbonl)oxy]-3-oxo-androsta-1,4-diene-17β-carbothioic acide *S*-fluoromethyl ester.